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IS 12706 (1989): Dye Intermediates - Naphthol AS-E [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard
DYE INTERMEDIATES — NAPHTHOL AS-E — SPECIFICATION

भारतीय मानक
रंजक मध्यक — नेफ्थोल एएस-ई — विशिष्टि

UDC 667·289 : 547·655·1

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1989

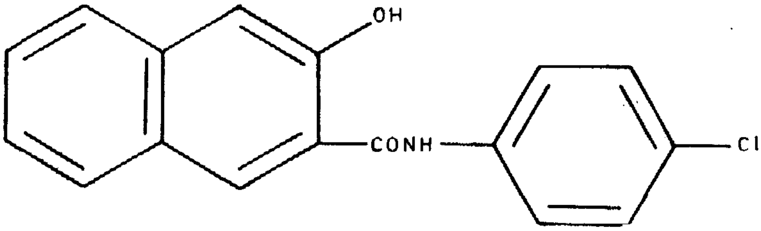
Price Group 2

Dye Intermediates Sectional Committee, PCDC 11

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 5 June 1989, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Naphthol AS-E (4-chloro-3-hydroxy-2-naphthanilide) or 2-naphthalene carboxamide finds extensive use in the manufacture of pigments due to its azoic coupling component, namely, N-(4-chloro-phanyl)-3-hydroxy group. It has the following structural formula:



NAPHTHOL AS-E

(4'-Chloro-3-Hydroxy-2-Naphthanilide)

CAS Registry Number [92-78-4]

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DYE INTERMEDIATES — NAPHTHOL AS-E — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for naphthol AS-E.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 4471 : 1981	Methods for determination of strength of naphthols (azoic coupling components) (gravimetric and volumetric method) (*first revision*)
IS : 5299 : 1969	Methods of sampling and tests for die intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of fine crystalline powder of light pink to light brown colour.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Naphthol AS-E

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Assay (on dry basis), percent by mass, *Min*	95·0	A-2
ii)	Free β-oxynaphthoic acid, percent by mass, *Max*	0·2	A-3
iii)	Matter insoluble in alkaline alcohol, percent by mass, *Max*	0·30	A-4

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer, and his recognized trade-mark, if any;

c) Batch number; and

d) Gross, net and tare mass.

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING

5.1 The material shall be sampled in accordance with **3** of IS : 5299 : 1969.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in Annex A, as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Table* 1 and *Clause* 6.1)

METHODS OF TEST FOR NAPHTHOL AS-E

A-1 PREPARED SAMPLE

A-1.1 Dry the material at 105 ± 1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests.

A-2 ASSAY

A-2.1 Assay in naphthol AS-E shall be estimated by the method as given in **4** or **5** of IS : 4471 : 1981.

A-3 DETERMINATION OF FREE β-OXYNAPHTHOIC ACID

A-3.0 General

Free β-oxynaphthoic acid content in naphthol AS-E is determined by thin layer chromatography method.

A-3.1 Apparatus

a) *Chromatographic Chamber,* 22 × 10 × 22 cm.

b) *Micropipette,* of 10 μl-capacity.

c) *Thin Layer Chromatographic Glass Plates,* 10 × 20 cm.

d) *Adjustable Thin Plate Applicator*

e) *Chromatographic Spray Bottle*

f) *Oven,* with a thermo regulator.

g) *Weighing Bottle*

A-3.2 Reagents

a) *β-Oxynaphthoic Acid*

b) *Methanol,* reagent grade,

c) *Fast Blue B Salt,* 0·1 percent solution,

d) *Eluent,* toluene : isopropanol : acetic acid (95:4:1) (*v/v*).

A-3.3 Procedure

A-3.3.1 *Preparation of the Chromatographic Plates*

Mix 50 g of neutral silica gel G with 85 to 90 ml of water for three minutes. Then, by means of the applicator, coat the plate with the silica gel layer of thickness not exceeding 0·25 mm and place it in the oven maintained at 110°C for 90 minutes. By means of a needle, mark a thin line width wise on all plates at about 3 cm from the upper edge. Also remove a band of about 5 mm width silica gel from either of the lateral sides of each plate.

A-3.3.2 *Preparation of the Sample and Reference Solutions*

A-3.3.2.1 Weigh about 2 g of the *prepared sample* (**A-1.1**) into a 50-ml test tube. Add 20 ml of methanol [**A-2.2** (b)] and mix well. Boil the contents of the test tube on a hot water bath for 2 minutes. Cool the contents of the test tube, filter and collect the filtrate in a stoppered bottle. Make up the volume to 20 ml with methanol.

A-3.3.2.2 Prepare solutions of β-oxynaphthoic acid by dissolving 10 mg, 20 mg, 30 mg and 40 mg of the material in 100 ml of methanol. This will provide 0·1 percent, 0·2 percent, 0·3 percent and 0·4 percent respectively of β-oxynaphthoic acid.

A-3.3.3 *Development of Chromatogram*

Spot 10 μl each of sample solution (**A-3.3.2.1**) and those of reference solutions of β-oxynaphthoic acid (**A-3.3.2.2**) on the thin layer plate. Place the eluent [**A-3.2** (d)] in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the eluent to run in ascending manner. Remove the plate from the chamber after the eluent has travelled 75 percent of the plate and dry off the solvent in an air drier. Spray the plate with the solution of fast blue B salt [**A-3.2**(c)]. Following spot characteristic of constituents are obtained:

Constituent	*Zone*	*Rf*
β-oxynaphthoic acid	I	0·30
Unknown impurity	II	0·40
Unknown impurity	III	0·44
Naphthol AS-E	IV	0·66
Unknown impurity	V	0·78

A-3.4 Report

Report β-oxynaphthoic content as that which is nearest in intensity to the reference sample. In case the colour intensity does not come in the range of the standard spots, repeat the whole procedure using suitable concentration of various impurities.

A-4 MATTER INSOLUBLE IN ALKALINE ALCOHOL

A-4.1 Reagents

a) *Sodium Hydroxide Solution,* 5 percent

b) *Alkaline Alcohol,* equal proportion of 5 percent sodium hydroxide alcohol.

A-4.2 Apparatus

a) *Sintered Glass G 4 or Gooch Crucible*

b) *Beaker,* 500 ml.

A-4.3 Procedure

Weigh accurately about 5 g of the *prepared sample* (**A-1.1**) into a 500-ml beaker. Add about 200 ml alcoholic sodium hydroxide solution (equal proportion of 5 percent sodium hydroxide solution and alcohol). Stir well and heat to dissolve the material completely. Add reagent, if necessary till the solution is alkaline to phenolphthalein indicator paper. Filter the solution through sintered glass G 4 or Gooch crucible. Wash the residue with water till washings are free from alkali. Dry the crucible at 100 ± 5°C till constant mass.

A-4.4 Calculation

$$\text{Matter insoluble in alkaline alcohol, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of residue, and

M_2 = mass in g of sample taken for test.

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